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L3: Entry 2 of 2

File: DWPI

Oct 9, 2002

DERWENT-ACC-NO: 2003-052373

DERWENT-WEEK: 200305

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TITLE: Manufacture of inorganic-type porous composite for use as filter or carrier material, involves performing phase transition in sol-gel reaction solution in which dispersed particle component co-exists

PATENT-ASSIGNEE:

ASSIGNEE

CODE

NAKANISHI K

NAKAI

SOGA N

SOGAI

PRIORITY-DATA: 2001JP-0103036 (April 2, 2001)

[Search Selected](#)[Search ALL](#)[Clear](#)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

PAGES

MAIN-IPC

☐ [JP 2002293657 A](#)

October 9, 2002

004

C04B038/00

APPLICATION-DATA:

PUB-NO

APPL-DATE

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DESCRIPTOR

JP2002293657A

April 2, 2001

2001JP-0103036

INT-CL (IPC): [B01 D 39/00](#); [B01 D 39/20](#); [C04 B 38/00](#)

ABSTRACTED-PUB-NO: JP2002293657A

BASIC-ABSTRACT:

NOVELTY - A dispersed particle component is made to co-exist in a sol-gel reaction solution. A phase transition is carried out in the sol-gel reaction solution so that an inorganic-type porous composite containing a structural component having open pores and dispersed particles is manufactured.

DETAILED DESCRIPTION - The dispersed particle is metallic oxide, metal, organic polymer or their composites.

An INDEPENDENT CLAIM is included for an inorganic-type porous composite comprising dispersed particles.

USE - For use as a filter or carrier material.

ADVANTAGE - The process forms an inorganic-type porous composite having a uniform

pore size distribution.

DESCRIPTION OF DRAWING(S) - The figure shows a graph representing the pore volume distribution of a porous composite with 0.25 g of alumina additional amount by mercury pressure process. (Drawing includes non-English language text).

CHOSEN-DRAWING: Dwg.1/2

TITLE-TERMS: MANUFACTURE INORGANIC TYPE POROUS COMPOSITE FILTER CARRY MATERIAL
PERFORMANCE PHASE TRANSITION SOL GEL REACT SOLUTION DISPERSE PARTICLE COMPONENT CO
EXIST

DERWENT-CLASS: J01 J04 L02

CPI-CODES: J01-H; J04-E03; L02-A02A;

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C2003-013611

[Previous Doc](#)

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L10: Entry 1 of 1

File: JPAB

Apr 22, 1991

PUB-NO: JP403096965A

DOCUMENT-IDENTIFIER: [JP 03096965 A](#)

TITLE: PRODUCTION OF POLYMERIZED TONER

PUBN-DATE: April 22, 1991

INVENTOR-INFORMATION:

NAME

COUNTRY

NAGASAWA, YUICHI

ASSIGNEE-INFORMATION:

NAME

COUNTRY

SANYO CHEM IND LTD

APPL-NO: JP01234922

APPL-DATE: September 11, 1989

US-CL-CURRENT: [430/137.17](#)

INT-CL (IPC): G03G 9/087; G03G 9/09

ABSTRACT:

PURPOSE: To sharpen the electrostatic charge quantity distribution of the toner and to obviate fogging as well as to increase image density by previously melting and kneading a carbon black with polyester to finely disperse the carbon black and dispersing this dispersion into monomers, then polymerizing the monomers.

CONSTITUTION: The carbon black (A) is previously melted and kneaded with the polyester (B) to finely disperse the component A to $\leq 0.2\mu\text{m}$ number average dispersion particle size. The finely dispersed component A is then dissolved and dispersed into the monomer (C) and is subjected to suspension polymn. to obtain the toner. The execution of emulsion polymn. is equally preferable. A styrene monomer and (meth)acrylic monomer and/or maleic acid monomer or monomer having an amino group are used for the component C and the carbon having $\geq 80\text{m}^2/\text{g}$ specific surface area is used for the component A. The polymer having the melt viscosity and glass transition point which are respectively in prescribed ranges is preferably used for the component B.

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特開2002-293657

(P2002-293657A)

(43) 公開日 平成14年10月9日 (2002.10.9)

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B 0 1 D 39/00		B 0 1 D 39/00	B
39/20		39/20	Z

審査請求 未請求 請求項の数 5 O L (全 4 頁)

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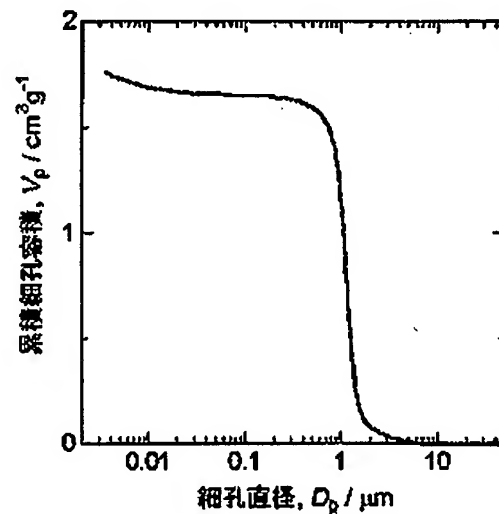
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(54) 【発明の名称】 分散粒子を含む無機系多孔質複合体の製造方法

(57) 【要約】

【課題】本発明は、バインダーを燃焼させることによるエネルギーコストと二酸化炭素排出による環境負荷を削減でき、しかも細孔形状やサイズ分布の均一な無機系多孔質複合体を製造することを目的とする。

【解決手段】本発明は、あらかじめ微粒子成分を分散させた溶媒中で網目形成成分の前駆体を反応させる。あるいは、あらかじめ網目形成成分を溶解した反応溶液に、微粒子分散液を混合して反応させることによって、相分離とゾル-ゲル転移を同時に引き起こし、直径100nm以上の気孔を有するゲルを形成させ、引き続いて湿潤ゲルの洗浄あるいは溶媒置換処理の後に、溶媒を除去し、必要に応じて適切な温度で熱処理することを特徴とする。



(2)

特開2002-293657

1

【特許請求の範囲】

【請求項1】ゾルーゲル反応溶液に分散粒子成分を共存させておき、相転移を伴うゾルーゲル転移を起こさせることによって開気孔と分散粒子を含む骨格成分からなる無機系多孔質複合体を製造することを特徴とする無機系多孔質複合体の製造方法。

【請求項2】開気孔が直径100nm以上である請求項1記載の無機系多孔質複合体の製造方法。

【請求項3】分散粒子が、金属酸化物、金属、有機高分子およびそれらの複合体である、請求項1又は2に記載の無機系多孔質複合体の製造方法。

【請求項4】分散粒子の平均直径が5nmから100μmである請求項1乃至3に記載の無機系多孔質複合体の製造方法。

【請求項5】骨格相に分散粒子を含み100nm以上の気孔を有する無機系多孔質複合体。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、分散粒子を含む無機系多孔質複合体の製造法に関する。本発明により製造された多孔質材料は、フィルターや担体材料として利用される。

【0002】

【従来の技術】一般にセラミックスに代表される無機系多孔質体は、バインダーあるいは造孔剤と呼ばれる樹脂成分によって結着された原料微粒子の圧縮成形体を、バインダーの焼結を伴って焼結させることにより作製されている。気孔の形成はバインダー焼失により、その占めていた空間が焼結粒子間に残ることによっており、細孔容積にほぼ等しい体積のバインダーを焼結させるエネルギーコストと二酸化炭素排出による環境負荷は極めて高い。また、粒子の連絡構造は必然的にネック部を有するため、細孔形状が不均一でそのサイズ分布も広いものとなることが多い。他方、相分離を利用したゾルーゲル法によって、シリカを始めとする無機系多孔質体が再現性良く製造されることが知られている。この方法では溶媒に富む相が蒸発することによって多孔構造が形成されるため、従来法よりも環境負荷を劇的に低減することができる。また、細孔形状やそのサイズ分布は極めて均一性が高く、従来よりも高効率な分離、精製プロセスを行うことができる可能性が高い。

【0003】

【発明が解決しようとする課題】そこで本発明者が研究したところ、シリカを主成分とするゾルーゲル反応溶液にあらかじめ分散粒子成分を共存させておき、粒子が沈降や凝集を起こさない条件で相分離を伴うゾルーゲル転移を起こさせることにより、ゲル相に分散粒子が取り込まれた多孔質複合体が得られることが明らかになった。

【課題を解決するための手段】すなわち、本発明は、ゾ

2

ルーゲル反応溶液に分散粒子成分を共存させておき、相転移を伴うゾルーゲル転移を起こさせることによって開気孔と分散粒子を含む骨格成分からなる無機系多孔質複合体を製造することを特徴とする無機系多孔質複合体の製造方法である。ここで、開気孔は、直径100nm以上、好ましくは200～1000nmである。直径100nm以上のマクロ孔は、相分離の際に生じる溶媒相の占めていた領域として形成されるので、通常の乾燥操作により燃焼や熱分解を伴うことなく形成し、溶媒相とゲル相が互々絡み合って連続したいわゆる共通構造を形成する場合に、極めて鋭いサイズ分布を得ることができる。

【0004】その方法は、あらかじめ微粒子成分を分散させた溶媒中で網目形成成分の前駆体を反応させる、あるいは、あらかじめ網目形成成分を溶解した反応溶液に、微粒子分散液を混合して反応させることによって、相分離とゾルーゲル転移を同時に引き起こし、直径100nm以上の気孔を有するゲルを形成させ、引き続き湿潤ゲルの洗滌あるいは溶媒置換処理の後に、溶媒を除去し、必要に応じて適切な温度で熱処理することを特徴とする。

【0005】相分離は、材料の製造プロセスにおいて、沈降や析出によって出発成分と異なる成分を持つ領域が生成する広汎な現象であり、ゾルーゲル反応系においては、ゲル形成を起こす網目形成成分に富む相（ゲル相）と、ゲル形成を起こさない溶媒成分に富む相（溶媒相）とに、分離が起こる。各相領域の形成にあたっては、化学ポテンシャルの差を駆動力として濃度勾配に逆らった成分の拡散が起こり、各相領域が与えられた温度・圧力下での平衡組成に達するまで、物質移動が継続する。この際に、出発組成に分散粒子成分を共存させ、なおかつ分散粒子成分が相分離やゾルーゲル反応に著しい影響を与えないような条件を選べば、分散粒子の性質に従って、ゲル相あるいは溶媒相に、優先的に分散粒子を分配することが可能となる。すなわち、ゲル相を形成する成分と親和性が高く溶媒相を形成する成分と親和性の低い分散粒子を共存させた場合には、分散粒子はゲル相に優先的に分配される。逆の場合には溶媒相に分散粒子が優先的に分配されることになるが、ゲル形成の後溶媒相を除去して多孔体を作製する場合には、前者の条件を満たす分散粒子を選択することが重要になる。

【0006】ゾルーゲル反応に用いられるゲル形成を起こす網目成分の前駆体としては、金属アルコキシド、錯体、金属塩、有機修飾金属アルコキシド、有機架橋金属アルコキシド、およびこれらの部分加水分解生成物、部分重合生成物である多量体を用いることができる。水ガラスはケイ酸塩水溶液のpHを変化させることによるゾルーゲル転移も、同様に利用することができる。

【0007】さらに本発明の具体的な製造方法は、水溶性高分子を酸性水溶液に溶かし、それに微粒子成分を分散させた後、加水分解性の官能基を有する金属化合物を

(3)

特開2002-293657

3

添加して加水分解反応を行い、生成物が固化した後、次いで乾燥し加熱する。ここで、水溶性高分子は、理論的には適当な濃度の水溶液と成し得る水溶性有機高分子であって、加水分解性の官能基を有する金属化合物によって生成するアルコールを含む反応系中に均一に溶解し得るものであれば良いが、具体的には高分子金属塩であるポリスチレンスルホン酸のナトリウム塩またはカリウム塩、高分子酸であって解離してポリアニオンとなるポリアクリル酸、高分子塩基であって水溶液中でポリカチオンを生ずるポリアリルアミンおよびポリエチレンイミン、あるいは中性高分子であって主鎖にエーテル結合を持つポリエチレンオキシド、あるいはポリビニルピロリドン等が好適である。また、有機高分子に代えてホルムアミド、多価アルコール、界面活性剤を用いてもよく、その場合多価アルコールとしてはグリセリンが、界面活性剤としてはポリオキシエチレンアルキルエーテル類が最適である。

【0008】加水分解性の官能基を有する金属化合物としては、金属アルコキシド又はそのオリゴマーを用いることができ、これらのものは例えば、メトキシ基、エトキシ基、プロポキシ基等の炭素数の少ないものが好ましい。また、その金属としては、最終的に形成される酸化物の金属、例えばS i、T i、Z r、A lが使用される。この金属としては1種又は2種以上であっても良い。一方オリゴマーとしてはアルコールに均一に溶解分散できるものであればよく、具体的には10畳体程度まで使用できる。また、これらのケイ素アルコキシドのアルコキシ基のいくつかはアルキル基に置換された、アルキルアルコキシシラン類、およびそれらの10畳体程度までのオリゴマーが好適に用いられる。またケイ素に代えて中心金属元素を、チタン、ジルコニウム、アルミニウム等に置換したアルキル置換金属アルコキシドも同様に用いることができる。

【0009】また、酸性水溶液としては、通常塩酸、硝酸等の鉱酸0.001規定以上のもの、あるいは辛酸、酢酸等の有機酸0.1規定以上のものが好ましい。加水分解にあたっては、溶液を室温40～80℃で0.5～5時間保存することによって達成できる。

【0010】現在工業的に生産・市販されている分散粒子は、有機高分子、金属酸化物あるいは金属を主成分とし、その粒径（平均直径）は5nm程度から100μm程度まで非常に広い範囲にわたっている。これらの微粒子と、ゲル形成を起こす網目成分との化学的な親和性は、多くの場合粒子表面の化学修飾などによって自由に制御できることが知られており、ゾル-ゲル反応時に凝集や沈降を起こさない条件を満たす粒子であれば、化学組成に関係なく本製造方法に適用することができる。したがって、本発明において分散粒子は、金属酸化物、金属、有機高分子およびそれらの複合体を用いることができ、好ましい平均直径は5nmから100μmである。具体的に

4

は、酸化ケイ素、酸化チタン、酸化ジルコニウム、酸化アルミニウム、酸化カルシウム、酸化マグネシウム、酸化鉄ほか遷移金属酸化物、酸化イットリウムおよび酸化ランタンほか希土類酸化物などが好適である。さらに反応溶液中で安定な炭酸塩、硝酸塩、硫酸塩、燐酸塩、ハロゲン化物、無機塩類なども同様に用いることができる。有機塩、錯体、保護された金属コロイド、高分子ラテックスほか微粒子状有機高分子も、反応溶液への分散性を制御することによって本発明による無機系多孔質複合体の作製に用いることができる。また、添加する分散粒子の量は、90重量%以下、好ましくは80重量%以下である。分散粒子の添加量の増加とともに細孔径は小さくなる。

【0011】本発明の製造方法によれば、倍格相に分散粒子を含み100nm以上の気孔を有する無機系多孔質複合体が得られる。

【0012】

【実施例】まず水溶性高分子であるポリエチレンオキシド（アルドリッチ製 商品番号85,645-2）0.90gを0.01規定酢酸水溶液10gに溶解し、この溶液にアルミナ粉末（平均粒径0.5ミクロン、住友化学工業（株）製 易焼結性アルミナ粉末 AES-12 #00601）をかくはん下で加えて分散させた。ついで、テトラメトキシシラン5mlをかくはん下で加えて、加水分解反応を行った。数分かくはんしたのち、得られた透明溶液を密閉容器に移し、40℃の恒温槽中に保持したところ約40分後に固化した。得られたゲルをそのままの温度で3日間熟成させ、そののち溶媒を蒸発除去することによって多孔質複合体を得た。アルミナ粉末の量を、0.25、0.50、1.0、2.00g（反応溶液に対して約1.5、3.0、5.9、11.1%）に変化させたところ、いずれの量においても連続貫通孔を持つ多孔質複合体が得られたが、アルミナ粉末の添加量の増加と共に細孔径は小さくなり、細孔の形状はアルミナを入れない場合に見られる棒状のものから、表面の粗い形状へと変化した。アルミナ添加量0.25gの多孔質複合体の細孔径分布を水銀圧入法で求めた結果を図1に示す。直径1.0ミクロンを中心とした鋭い細孔分布が得られていることが分かる。また、アルミナ添加量0.50gの多孔質複合体の細孔径分布を水銀圧入法で求めた結果を図2に示す。直径0.7ミクロンを中心とした鋭い細孔分布が得られていることが分かる。アルミナ粉末の添加量の増加と共に細孔径は小さくなる。

【0013】

【発明の効果】本発明によれば、従来法のようなバインダーを焼結させることによるエネルギーコストと二酸化炭素排出による環境負荷を削減でき、しかも細孔形状やサイズ分布の均一な無機系多孔質複合体を製造できる。

【図面の簡単な説明】

【図1】アルミナ添加量0.25gの多孔質複合体の細孔径分布を水銀圧入法で求めた図

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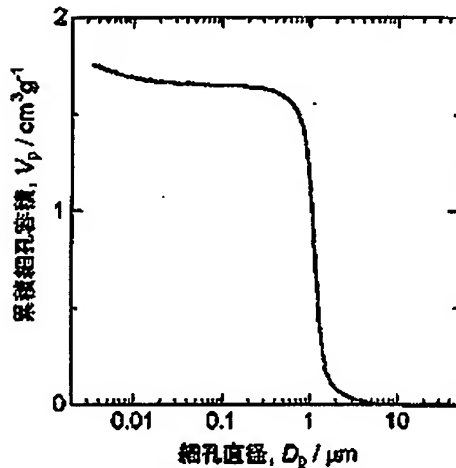
特開2002-293657

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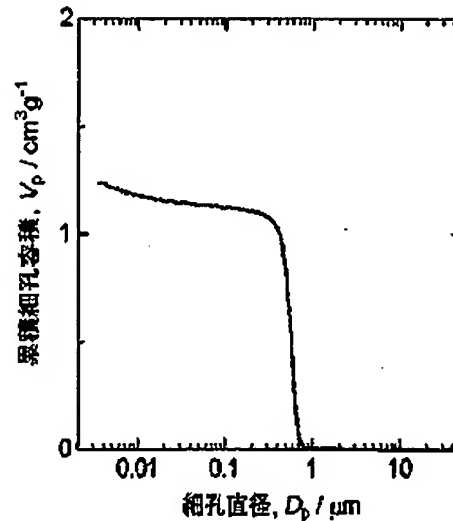
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【図2】アルミナ添加量0.50gの多孔質複合体の細孔径 * * 分布を水銀圧入法で求めた図

【図1】



【図2】



【手続補正音】

【提出日】平成13年5月31日(2001.5.31)

【手続補正1】

【補正対象書類名】明細書

【補正対象項目名】請求項1

【補正方法】変更

【補正内容】

【請求項1】ゾルーゲル反応溶液に分散粒子成分を共存させておき、相分離を伴うゾルーゲル転移を起こさせることによって開気孔と分散粒子を含む骨格成分からなる無機系多孔質複合体を製造することを特徴とする無機系多孔質複合体の製造方法。

【手続補正2】

【補正対象書類名】明細書

【補正対象項目名】0003

【補正方法】変更

【補正内容】

【0003】

【発明が解決しようとする課題】そこで本発明者が研

究したところ、シリカを主成分とするゾルーゲル反応溶液にあらかじめ分散粒子成分を共存させておき、粒子が沈降や凝集を起こさない条件で相分離を伴うゾルーゲル転移を起こさせることにより、ゲル相に分散粒子が取り込まれた多孔質複合体が得られることが明らかになった。

【課題を解決するための手段】すなわち、本発明は、ゾルーゲル反応溶液に分散粒子成分を共存させておき、相分離を伴うゾルーゲル転移を起こさせることによって開気孔と分散粒子を含む骨格成分からなる無機系多孔質複合体を製造することを特徴とする無機系多孔質複合体の製造方法である。ここで、開気孔は、直径100nm以上、好ましくは200～1000nmである。直径100nm以上のマクロ孔は、相分離の際に生じる溶媒相の占めていた領域として形成されるので、通常の乾燥操作により燃焼や熱分解を伴うことなく形成し、溶媒相とゲル相が互々絡み合って連続したいわゆる共連続構造を形成する場合には、極めて鋭いサイズ分布を得ることができる。

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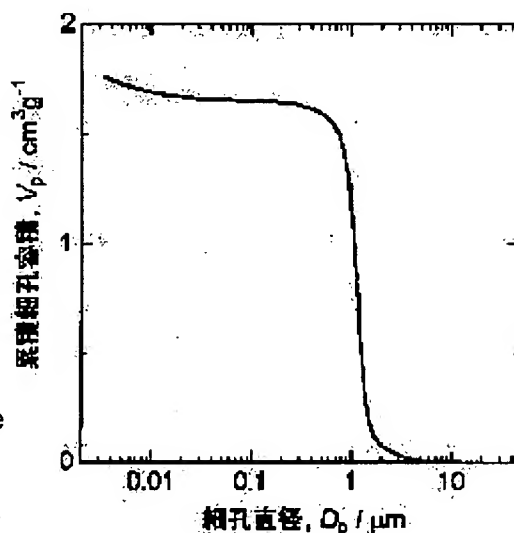
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NAKANISHI KAZUKI(54) METHOD OF MANUFACTURING INORGANIC POROUS COMPOSITE CONTAINING
DISPERSED PARTICLE

(57)Abstract:

PROBLEM TO BE SOLVED: To manufacture an inorganic porous composite whose energy cost to be raised by burning a binder and whose environmental load to be increased by discharging carbon dioxide, can be cut down and whose pore shape and pore size distribution are made uniform.

SOLUTION: This inorganic porous composite is manufactured by forming the gel having the pores of ≥ 100 nm diameter by reacting a fine particle component with a precursor of a mesh forming component in the fine particle component-dispersed solvent or mixing the fine particle-dispersed liquid in the mesh forming component-dissolved reaction solution to react them so that phase separation and sol-gel transition are caused at the same

time, cleaning the wet gel with a new solvent or displacing the solvent of the wet gel with the new solvent, removing the new solvent and heat-treating the solvent-removed gel at the adequate temperature, if necessary.



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CLAIMS

[Claim(s)]

[Claim 1] The manufacture approach of the inorganic system porosity complex characterized by manufacturing the inorganic system porosity complex which consists of an open pore and a frame component containing a particulate material by making the particulate material component live together in a sol-gel reaction solution, and making the sol-gel transition accompanied by phase transition cause.

[Claim 2] The manufacture approach of inorganic system porosity complex according to claim 1 that an open pore is the diameter of 100nm or more.

[Claim 3] The manufacture approach of inorganic system porosity complex according to claim 1 or 2 that particulate materials are a metallic oxide, a metal, organic macromolecules, and those complex.

[Claim 4] The manufacturing method of the inorganic system porosity complex according to claim 1 to 3 whose average diameter of a particulate material is 100 micrometers from 5nm.

[Claim 5] Inorganic system porosity complex which has pore 100nm or more including a particulate material in a frame phase.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the manufacturing method of the inorganic system porosity complex containing a particulate material. The porous material manufactured by this invention is used as a filter or a support ingredient.

[0002]

[Description of the Prior Art] The inorganic system porous body generally represented by the ceramics is produced by making the compression-molding object of a raw material particle bound by the resinous principle called a binder or an ostomy agent sinter with combustion of a binder. The environmental load by the energy cost and carbon dioxide emission which it gets down [carbon dioxide emission] from formation of pore by binder destruction by fire when the occupied space remains between sintering particles, and burn the binder of the volume almost equal to pore volume is very expensive. Moreover, since the connection structure of a particle has the neck section inevitably, its pore configuration is uneven and it will become large [the size distribution] in many cases. On the other hand, it is known by the sol-gel method using phase separation that inorganic system porous bodies including a silica will be manufactured with sufficient repeatability. By this approach, since porous structure is formed when the phase which is rich in a solvent evaporates, an environmental load can be dramatically reduced rather than a conventional method. Moreover, a pore configuration and its size distribution have very high homogeneity, and its possibility that separation more efficient than before and a refinery process can be performed is high.

[0003]

[Problem(s) to be Solved by the Invention] Then, when this artificer inquired, it became clear that the porosity complex with which the particulate material was incorporated by the gel phase is obtained by making the particulate material component live together beforehand in the sol-gel reaction solution which uses a silica as a principal component, and making the sol-gel transition accompanied by phase separation cause on the conditions from which a particle starts neither sedimentation nor condensation.

[Means for Solving the Problem] That is, this invention is the manufacture approach of the inorganic system porosity complex characterized by manufacturing the inorganic system porosity complex which consists of an open pore and a frame component containing a particulate material by making the particulate material component live together in a sol-gel reaction solution, and making the sol-gel transition accompanied by phase transition cause. Here, an open pore is 200-10000nm preferably the diameter of 100nm or more. Since macropore with a diameter of 100nm or more is formed as a field which occupied the solvent phase produced in the case of phase separation, when forming without being accompanied by combustion or the pyrolysis by the usual desiccation actuation and forming the so-called co-continuous structure with which the solvent phase and the gel phase became entangled respectively and which they followed, it can obtain a very sharp size distribution.

[0004] The approach makes the precursor of the network former react in the solvent which distributed the particle component beforehand. Or by making particle dispersion liquid mix and react to the reaction

solution which dissolved the network former beforehand Cause phase separation and sol-gel transition simultaneously, the gel which has pore with a diameter of 100nm or more is made to form, a solvent is succeedingly removed after washing of humid gel, or solvent permutation processing, and it is characterized by heat-treating at suitable temperature if needed.

[0005] Phase separation is an extensive phenomenon which the field which has a start component and a different component by precipitate or deposit generates in the manufacture process of an ingredient, and separation takes place to the phase (gel phase) which is rich in the network former which causes gel formation, and the phase (solvent phase) which is rich in the solvent component which does not cause gel formation in the sol-gel system of reaction. In formation of each phase field, diffusion of the component which opposed the concentration gradient by making the difference of chemical potential into driving force takes place, and mass transfer continues until it reaches the equilibrium composition under the temperature and the pressure to which each phase field was given. under the present circumstances, it is alike and a particulate material component is together lived in a start presentation -- making -- in addition -- and if conditions which do not have effect with a particulate material component remarkable for phase separation or a sol-gel reaction are chosen, according to the property of a particulate material, it will become possible to distribute a particulate material to a gel phase or a solvent phase preferentially. Namely, when a particulate material with low component which forms a gel phase, component in which compatibility forms a solvent phase highly, and compatibility is made to live together, a particulate material is distributed with the priority to a gel phase. Although a particulate material will be preferentially distributed to a solvent phase at the case of reverse, in removing a solvent phase after gel formation and producing a porous body, it becomes important to choose the particulate material which fulfills the former conditions.

[0006] As a precursor of the mesh component which causes the gel formation used for a sol-gel reaction, the polymer which are a metal alkoxide, a complex, a metal salt, an organic qualification metal alkoxide, organic bridge formation metal alkoxides and these partial hydrolysis products, and a partial polymerization product can be used. The sol-gel transition by changing pH of a silicate water solution besides water glass can be used similarly.

[0007] Furthermore, after adding the metallic compounds which have the functional group of hydrolysis nature after the concrete manufacture approach of this invention melting a water soluble polymer to aqueous acids and making it distribute a particle component, performing a hydrolysis reaction and a product's solidifying, subsequently it dries, and it heats. Water soluble polymers are the water solution of suitable concentration, and a water-soluble organic macromolecule which can be accomplished theoretically here. Be [what is necessary / just although it may dissolve in homogeneity into the system of reaction containing the alcohol generated with the metallic compounds which have the functional group of hydrolysis nature] Sodium salt or potassium salt of polystyrene sulfonate which is specifically a giant-molecule metal salt, The polyacrylic acid which is a macromolecule acid, dissociates and serves as the poly anion, the poly allylamine which is a macromolecule base and produces the poly cation in a water solution, and polyethyleneimine, Or the polyethylene oxide which is a neutral giant molecule and has ether linkage in a principal chain, or a polyvinyl pyrrolidone is suitable. Moreover, it may replace with an organic macromolecule, a formamide, polyhydric alcohol, and a surfactant may be used, and the glycerol of polyoxyethylene alkyl ether is the optimal in that case as a surfactant as polyhydric alcohol.

[0008] As metallic compounds which have the functional group of hydrolysis nature, a metal alkoxide or its oligomer can be used and, as for these things, what has few carbon numbers, such as a methoxy group, an ethoxy radical, and a propoxy group, is desirable. Moreover, as the metal, the metal of the oxide formed eventually, for example, Si, Ti, Zr, and aluminum, is used. As this metal, you may be one sort or two sorts or more. On the other hand as oligomer, it can specifically be used for alcohol to 10 **** extent at homogeneity that what is necessary is just what can carry out dissolution distribution. Moreover, the oligomer to the alkyl alkoxysilane by which some of alkoxy groups of these silicon alkoxides were permuted by the alkyl group, and those 10 **** extent is used suitably. Moreover, the alkylation metal alkoxide which changed into silicon and permuted the main metallic element by titanium, the zirconium, aluminum, etc. can be used similarly.

[0009] Moreover, as aqueous acids, the thing more than organic-acid decinormals, such as a thing of 0.001 or more ****s of mineral acids, such as a hydrochloric acid and a nitric acid, or a formic acid, and an acetic acid, is usually desirable. In hydrolysis, it can attain by saving a solution at the room temperature of 40-80 degrees C for 0.5 to 5 hours.

[0010] The particulate material produced and marketed by the current industrial target uses an organic macromolecule, a metallic oxide, or a metal as a principal component, and the particle size (average diameter) is crossed to the range very large from about 5nm to about 100 micrometers. It is known that it will be freely controllable by the chemical modification of many field granulation child front faces etc., and if the chemical compatibility of these particles and the mesh component which causes gel formation is a particle which fulfills the conditions which cause neither condensation nor sedimentation to sol-gel reaction time, it is applicable to this manufacture approach regardless of chemical composition.

Therefore, in this invention, a metallic oxide, a metal, organic macromolecules, and those complex can be used for a particulate material, and a desirable average diameter is 100 micrometers from 5nm.

Specifically, silicon oxide, titanium oxide, a zirconium dioxide, an aluminum oxide, a calcium oxide, a magnesium oxide, a transition-metals oxide besides an iron oxide, yttrium oxide, a rare earth oxide besides a lanthanum trioxide, etc. are suitable. The still more stable carbonate in a reaction solution, a nitrate, a sulfate, phosphate, a halogenide, mineral, etc. can be used similarly. A particle-like organic macromolecule besides organic salt, a complex, the protected metal colloid, and a macromolecule latex can also be used for production of the inorganic system porosity complex by this invention by controlling the dispersibility to a reaction solution. Moreover, the amount of the particulate material to add is 80 or less % of the weight preferably 90 or less % of the weight. Pore size becomes small with the increment in the addition of a particulate material.

[0011] According to the manufacture approach of this invention, the inorganic system porosity complex which has pore 100nm or more including a particulate material in a frame phase is obtained.

[0012]

[Example] Polyethylene oxide (Aldrich goods number 85,645-2) 0.90g which is a water soluble polymer first was dissolved in 10g of centinormal acetic-acid water solutions, and this solution was made to, distribute alumina powder (mean particle diameter of 0.5 microns, sinterable alumina powder by Sumitomo Chemical Co., Ltd. AES-12 #00601) in addition under stirring. Subsequently, it is under stirring about tetramethoxy silane 5ml, in addition the hydrolysis reaction was performed. After agitating several minutes, the obtained transparence solution was moved to the well-closed container, and when held in the 40-degree C incubator, it solidified after about 40 minutes. The obtained gel was ripened for three days at temperature as it is, and massive porosity complex was obtained by carrying out evaporation clearance of the after solvent. Although the porosity complex which has a continuation breakthrough also in which amount was obtained when the amount of alumina powder was changed to 0.25, 0.50, and 1.00 and 2.00g (they are about 1.5, 3.0, and 5.9 or 11.1% to a reaction solution) Pore size became small with the increment in the addition of alumina powder, and the configuration of pore changed from the smooth thing seen when not putting in an alumina to the configuration where a front face is coarse. The result of having searched for the pore volume distribution of porosity complex with an alumina addition of 0.25g with the method of mercury penetration is shown in drawing 1 . It turns out that sharp pore distribution centering on the diameter of 1.0 microns is acquired. Moreover, the result of having searched for the pore volume distribution of porosity complex with an alumina addition of 0.50g with the method of mercury penetration is shown in drawing 2 . It turns out that sharp pore distribution centering on the diameter of 0.7 microns is acquired. Pore size becomes small with the increment in the addition of alumina powder.

[0013]

[Effect of the Invention] According to this invention, the environmental loads by the energy cost by burning a binder like a conventional method and carbon dioxide emission can be reduced, and, moreover, the uniform inorganic system porosity complex of a pore configuration or a size distribution can be manufactured.

[Translation done.]